PATENT SPECIFICATION

(11) 1 379 707

1 379 707

(21) Application No. 10099/73 (22) Filed 1 March 1973

(31) Convention Application No. 21253 (32) Filed 1 March 1972 in

(33) Italy (IT)

(44) Complete Specification published 8 Jan. 1975

(51) INT CL² A23L 3/00; A23B 9/00

(52) Index at acceptance A2D 2D1 2D2 3B4X

(72) Inventor ENRICO SHEJBAL

(19)

(54) METHOD AND APPARATUS FOR PRESERVING PERISHABLE MATERIAL

(71) We, SNAM PROGETTI S.P.A., an Italian Company, of Corso Venezia, 16, Milan, Italy, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to a method and apparatus for preserving perishable material.

More particularly, but not exclusively, the present invention relates to a method for preserving cereals and valuable seeds stored in silos or like containers.

The preservation of wheat and cereals after harvesting, has always been a serious problem which has been dealt with in many different ways.

It is well known, however, that after harvesting and thrashing, the preservation of cereals cannot generally exceed 30 days unless the cereals have been previously and suitably treated. The present custom is to preserve the cereals in vertical silos for economical reasons relating, for example, to the ease of loading and unloading, and to the saving of space.

Such a known method suffers from several disadvantages among which the following may be mentioned:

(a) the preservation in silos requires a very low moisture content of the wheat, never higher than 14% for cereals since beyond this limit there may appear in a short time a plentiful fungus or bacterial flora capable of significantly altering the organoleptic properties of the ensiled material;

(b) the grains, even when ensiled, retain a light breathing power which causes in the deeper layers of the silo a significant increase of temperature; because of this phenomenon, water evaporates from the warmer deep layers and condenses in the surface layers; therefore the grains present in the upper regions of the silo can reach high moisture content percentages thereby providing the optimum conditions for fungi to develop; thus there is produced the harmful "outcrop";

(c) in actual practice the use of totally airtight silos is not easy to achieve, and this permits the entry and growth of insects which are difficult to control; in order to limit the damage caused by insects in the ensiled products, use is made at present of a large variety of insecticides by means of which it is possible to reduce or eliminate infestation before or after the storing or during the preserving period; however, it is known that these substances are very toxic and may provoke serious damages during the treatment or by accumulation in the caryopses; and

(d) the storage in large silos has until now resulted in a significant loss of germinability, which represents a serious disadvantage for valuable seeds.

It will thus be apparent that the preservation of cereals in silos is complex; it has required plant for controlling and regulating the temperature, for ventilating the silos and for transporting and mixing the wheat or other cereal inside the silos.

Further methods are known for storing perishable materials by making use of artificial environments, like those, for example, for preserving fresh fruits and vegetables. However, these methods either are not convenient in practice, since the preserving involves previous sealing of the containers thereby preventing the sampling and thereby maintaining at the same time the remaining part under the action of the inert environment; or make use of certain gases, for example CO_2, which cause serious germinability alterations (J. Le Du. Industr. Alim. Agr. *85* 811—821, 1968).

According to one aspect of the present invention, there is provided a method for preserving perishable material, which comprises passing through the perishable material a stream of nitrogen having a relative humidity in the range from 45% to 70%; monitoring the humidity of the stream of nitrogen after its passage through the perishable material; and controlling, in response to the monitored humidity, the relative humidity of the stream of nitrogen before its passage through the perishable material.

The method of the present invention makes

[Pric- -- ·

possible the sampling of the perishable material being stored, without causing the remaining part of the material to lose the benefit of the inert environment.

The method of the present invention prevents the growth and reproduction of parasitical insects and of their larvae without the use of toxic insecticides, allows a simpler storage of cereals for semination and an easy control of the moisture content of the perishable material, and eliminates the need for expensive treatments for preventing the growth of moulds which are eliminated together with their toxins. Many Aspergilli may determine the production of aphlatoxins, substances which are known for their carcinogenic activity and which are particularly toxic for domestic animals and man.

In the method of the present invention, the nitrogen used may be totally pure (apart from moisture) or may contain small amounts of oxygen or inert gases. The oxygen content should not exceed 2% by weight, and other gases which are inert to the perishable material, like CO and CO_2, should not be present in a quantity of more than 6% by weight based on the quantity of nitrogen.

An oxygen content higher than 2% by weight can cause serious drawbacks, by influencing the survival rate of insects which can exist even in the presence of comparatively low quantities of oxygen.

The nitrogen used must be suitably humid so as to keep unaltered the germinability properties of the ensiled products, which properties are very important if the products are designed for sowing, if a reduction in the yield of the ensiled products is to be avoided. This is very important if seeds of a valuable sort or species are being stored. Thus, as mentioned above, the relative humidity must lie in the range from 45% to 70%. Outside this range, serious drawbacks can occur, for example serious weight losses if the nitrogen is very dry, or a quick growth of moulds in the ensiled materials if the nitrogen is too humid, especially if the ensiled products are no longer in the original anaerobic or substantially anaerobic condition.

The method of the present invention is of general application; thus it may be applied for storing many different types of perishable material, but it is particularly suitable for storing cereals and valuable seeds.

Another aspect of the present invention provides an apparatus which comprises a container for accommodating the perishable material, means for introducing a stream of gaseous nitrogen into an upper region of the container, means allowing the escape of the stream of gaseous nitrogen from a lower region of the container, monitoring means for monitoring the humidity of the escaping stream of nitrogen, and means responsive to said monitoring means for controlling relative humidity of the stream of nitrogen introduced into the container.

The container is conveniently a silo which is sealed. The outlet point for the gas stream is located in a lower region of the silo and the inlet is located in the upper region; the gas outlet in the lower region is preferably beneath the column of the ensiled product. The humidity of the influent gas can be suitably regulated so as to keep it within the above-mentioned limits.

For a better understanding of the present invention and to show how the same may be carried into effect, reference will now be made, by way of example, to the accompanying drawings, in which:

Figure 1 represents diagrammatically a silo and associated equipment for carrying out the method of the present invention;

Figure 2 shows graphically the similarity in germinability between wheat stored in the silo with an air flow and wheat stored in the silo with a flow of technical nitrogen containing less than 0.5% by weight of oxygen, the silos being those referred to in the following Example;

Figure 3 shows graphically the moisture content of the wheat in the silo with an air flow and that of the wheat in the silo with a technical nitrogen flow; and

Figure 4 shows graphically the temperature inside wheat in the silo with an air flow and the temperature inside wheat in the silo with a technical nitrogen flow.

Referring firstly to Figure 1, there is shown a container 7, in this case a silo, for storing perishable material. There is also shown a pipe 1 for supplying nitrogen, which may be fed in the gaseous state or in the regasified state (starting from a liquid one). The pipe 1 is in communication with a source of nitrogen (not shown), for example a gas bottle or an exhaust of a system which has used the same nitrogen, *e.g.* from a refrigeration unit.

The pipe 1 leads to a two-way valve 2 which communicates either with a pipe 3 leading to a humidifier 4, or directly with a pipe 5. The pipe 5 leads to a pipe 6 leading directly to an upper region of the container 7. The pipe 3 passes through the humidifier 4 and leads to the pipe 5. At the lower end region of the container 7 is a discharge pipe 8 in which is situated a monitoring device 9 which is linked to the two-way valve 2. The two-way valve 2 is responsive to the device 9 and is hence dependent on the outlet humidity. The valve 2 determines whether the nitrogen in pipe 1 is passed through the humidifier 4, and what the relative humidity of the nitrogen entering the container 7 should be.

If the device 9 detects too high a relative humidity, then the nitrogen in pipe 1 is sent to the container 7 directly through valve 2

and pipes 5 and 6 without passing through pipe 3 and the humidifier 4. Suitable gas flow regulators (not shown) can be present in the nitrogen path.

The results obtained when storing wheat for eight months in a small silo under a continuous flow of so-called technical nitrogen, which had been suitably moistened, showed that, as a result of the treatment, no variation in germinability and in protein content of the wheat occurred; moreover, there were all the advantages of ventilation and asphyxia because during the whole storage period there was no development either of mould or of insects inside the silo.

Furthermore, laboratory tests have shown that the simulated wheat infection of the commonest parasitic insects was easily and totally eliminated in a short time, this being due to the fact that the passage of a continuous nitrogen stream through the silo produces uninterrupted asphyxia which eliminates any parasitic insects.

Our results have further shown that by ventilating the wheat with suitably moistened nitrogen it is possible to regulate easily the moisture content of the wheat and hence its weight, without any danger of mould.

EXAMPLE 1.

In order to test wheat storage in a controlled environment, two small cylindrical silos having an internal diameter of 50 cm and an internal height of 250 cm were manufactured.

Each cylinder was made of an asbestos-cement known as "Eternit" and each had a wall thickness of 1.75 cm. The word "Eternit" is a registered Trade Mark.

Each silo had at its top a cover and at its bottom a base, each of the cover and base being in the form of a metal plate sealing the cylinder with rubber gaskets and an adhesive based on cellulose. At three points, namely at 70 cm, 130 cm and 190 cm from the bottom of the silo, there were provided wheat sampling devices each consisting of a valve (Ø 15", gas mixture) having a stainless steel ball and a chromium-plated bronze body with gas-proof "Teflon" gaskets. The word "Teflon" is a registered Trade Mark. At points at the same heights there were thermometers for measuring the temperature on the axis of the cylinder. In a lower region of each silo there was an outlet in the form of a metal chute with a teflon-tight closure. The wheat column was supported on a stainless steel net arranged 20 cm above the silo bottom. A gas inlet was located in an upper region of each silo 15 cm below the cover, and a gas outlet in a lower region, beneath the wheat column, 10 cm above the base. The asbestos-cement cylinder and the A—CO iron plates were varnished with a synthetic oil and synthetic lacquer varnish.

The humidity of the gas being sent to the silo was adjusted by means of a system which permitted mixing of a high humidity gas, obtained by bubbling the gas through a column of water, with a dry gas. The humidity of each of the influent and effluent gases was continuously measured with a recording thermohygrometer (manufactured by Salmoiraghi S.p.A., of Rome, Italy) and the humidity of the influent gas was manually regulated by means of suitable valves. The flow of each of the influent and effluent gases was measured with low-flow flowmeters (0.5 to 10 litres/hour, sold by Asa of Monza).

The wheat used in the storage test in a controlled environment was soft wheat, cultivar *Conte Marzotto*, cultivated in Monterotondo and sacked soon after thrashing without being subjected to any treatment. The moisture content of the wheat at the time of harvesting was 10.06%.

The gases, to be moistened as hereinafter described, to be passed through the two silos, one gas through one silo and the other gas through the other silo, were as follows:—

(a) technical nitrogen, delivered by the Company SIO, of Rome, in the form of liquefied gas, declared oxygen concentration less than 0.5% by weight, regasified and warmed to room temperature;

(b) compressed-air having a low relative humidity (<30%).

The silos were loaded with wheat through the cover (about 450 kg/silo) and then sealed. Use has been made of the nitrogen and air stream, respectively, at a rate of from 4 to 8 litres per hour, and at a relative humidity of from 45 to 70%; 55% was the mean value (extreme values of 30 and 90% were admitted only for periods not exceeding 24 hours).

In addition to the continuous recording of the temperature and humidity either of the environment or of the influent and effluent gases, there were recorded periodically (four times per day) the influent and effluent gas flow and the temperature inside the silos at the levels of the wheat sampling.

At predetermined periods (mainly twice per week and at least once every three weeks), samples were taken from inside the wheat mass at the three sampling points at three heights. The measurement of the moisture content of the wheat was carried out by drying the samples in a stove at 150° C and in a drier at room temperature until constant weight was obtained; and the measurement of germinability in 4 day tests at room temperature was carried out by taking into account the germinability on 100 wheat grains from each sample.

The wheat protein content was analysed, and tests were carried out on the survival of wheat parasitic insects in inert environments. The methods used are reported hereinafter

together with the results obtained.

Figure 2 shows the germinability of wheat stored in the silo with an air flow (curve 1) and that of wheat stored in the silo with a flow of technical nitrogen containing less than 0.5% by weight of oxygen (curve 2) for a period of 8 months. After initial values lower than 90%, the germinability assumed in both cases a mean value of about 96% corresponding to the germinability of the same wheat when stored in ventilated sacks. The germinability is shown on the ordinates, and the time (in weeks) on the abscissae. As can be seen, there is no significant difference between the germinability of the wheat in the two silos.

Figure 3 shows on the ordinates the moisture content of wheat in the silos and on the abscissae the time in weeks. Curve 1 refers to the silo with an air flow and curve 2 to the silo with the technical nitrogen flow. The wheat humidity increased during the storage from the initial value of 10.06% to a value of about 11%.

During the whole storage period, the contents of the silos showed neither mould growth nor macroscopic modifications of the wheat. The moulding of the wheat during the germinability test did not exceed 2%, which indicates a very low contamination of the stored wheat.

Tests with small amounts of wheat in containers with a nitrogen flow having a high relative humidity (95%) showed that it is possible for the moisture content of the wheat to be as high as 20% without causing mould growth provided the wheat is for a limited period of time in asphyxiated conditions.

During the whole storage of the wheat, the temperature inside the silos only occasionally (because of thermal inertia) reached maximum values slightly higher than those of the environment in which they were situated, indicating thereby a sufficient ventilation of the wheat and the absence of overheating. In Figure 4 the temperature is indicated on the ordinates and the time in weeks on the abscissae. Curve 1 refers to the silo with an air flow and curve 2 to the silo with the nitrogen flow. The environmental temperature is shown in each of Curves 1 and 2 as a broken line. After four months from the beginning of the storage, an analysis was carried out of the protein content of the wheat stored under the air flow and the nitrogen flow. Use was made of the electrophoretic semi-quantitative method on acrylamide gel specified by Silano et al. (V. Silano, U. De Cellis, R. Deponte, A. M. D'errico, F. Pocchiari: Ric. Sci. *38*, 745 (1968)). No difference in the content of albumins, gliadins and globulins in the wheat stored in the two silos was observed.

During the whole storage period of the wheat, no spontaneous contamination due to wheat parasitic insects was observed. In order to produce such an infection and to study the effect of the gases passed through the silos, the survival rates of two species of insects generally present in Italian granaries, namely *Sitophilus granarius* and *Tribolium confusum*, were investigated.

The insects (10 per test, with some wheat grains in order to facilitate their movement) were put in air-tight glass containers and kept under a continuous flow of the following gases:

(a) air leaving the silos as a stream of humidified air;

(b) nitrogen leaving the silos as a stream of humidified technical nitrogen;

(c) humidified high grade nitrogen (<5 ppm of oxygen).

The results were as follows:—

(a) the survival of the insects of both species in the effluent air from the silo was 100% for the whole period of the test (10 days). Moreover, the behaviour of the insects was normal;

(b) in the initial period of the treatment with nitrogen leaving the silo, an acceleration of the movements of the insects in both species was noticed for about 2 hours. At the end of this period the *T. confusum* came into the comatose state, whereas the *S. granarius* continued to move slowly and came into the comatose state only after about 24 hours. As shown in the following Table 1, the survival of the insects of both species was very different. The LD (lethal dose) 100 at room temperature (20° C) for the *T. confusum* was reached after 24 hours in technical nitrogen, whereas for the *S. granarius* 120 hours were required.

(c) the behaviour of the insects in the completely asphyxiating environment of high grade nitrogen at a temperature of 20° C was similar to that in technical nitrogen, except that the induction times of the comatose state and for reaching LD 100 were shorter (as is clear from Table 1).

We have carried out a test of wheat preservation in conditions of complete asphyxia, high relative humidity and room and elevated temperatures. A small amount of wheat was preserved for 3 months under a high flow (10 l/h) of high grade nitrogen (<5 ppm O_2), humidified at 95%, at two temperatures: room temperature (about 20° C) and 40 ± 2° C.

TABLE 1

Survival rate of *T. confusum* and *S. granarius* in technical nitrogen or high grade nitrogen

Stay time in controlled environment (hours)	Survival (%)					
	T. confusum			*S. granarius*		
	air	N_2 tec. (< 0.5% by weight O_2)	high grade N_2 (< 5 ppm O_2)	air	N_2 tec (< 5% by weight O_2)	N_2 (high grade) (< 5 ppm O_2)
2	100	100	100	100	100	100
8	100	60	40	100	100	100
16	100	20	20	100	100	100
24	100	0	0	100	100	100
48	100	0	0	100	100	80
72	100	0	0	100	60	40
96	100	0	0	100	20	0
120	100	0	0	100	0	0

The following Table 2 shows the characteristics of wheat preserved under completely asphyxiating conditions for three months, namely high grade nitrogen, and relative humidity of 95%, at two temperatures:—

TABLE 2.

Temperature	Germinability (%)	Moisture Content (%)
Untreated control	97	10.6
Room (20° C)	96	19.9
40 ± 2° C	97	9.5

As can be seen from Table 2, no decrease in the wheat germinability was observed, and although the moisture content attained was 19.9% and 9.5% respectively, there was no moulding of the wheat at both the temperatures used.

WHAT WE CLAIM IS:—

1. A method for preserving perishable material, which comprises passing through the perishable material a stream of nitrogen having a relative humidity in the range from 45% to 70%; monitoring the humidity of the stream of nitrogen after its passage through the perishable material; and controlling, in response to the monitored humidity, the relative humidity of the stream of nitrogen before its passage through the perishable material.

2. A method according to Claim 1, wherein the stream of nitrogen contains oxygen in an amount of less than 2% by weight of the nitrogen.

3. A method according to Claim 2, wherein the stream of nitrogen contains less than 0.5% by weight of oxygen, based on the nitrogen.

4. A method according to Claim 3, wherein the stream of nitrogen contains less than 5 parts per million of oxygen, based on the nitrogen.

5. A method according to any preceding claim, wherein the nitrogen stream contains another gas inert to the perishable material, in an amount of not more than 6% of the nitrogen.

6. A method according to Claim 5, wherein the other gas inert to the perishable material is selected from carbon dioxide and carbon monoxide.

7. A method according to Claim 1, wherein the stream of nitrogen consists of pure nitrogen, apart from moisture.

8. A method according to any preceding claim, which also includes storing the perishable material in a silo, tightly closing the silo, introducing the stream of nitrogen into an upper region of the column of the stored material in the silo and allowing the stream of nitrogen which has passed through the column to be discharged at a lower region of the silo.

9. A method according to any preceding claim, wherein the perishable material is cereal grain or seed.

10. A method according to Claim 1, substantially as described in the foregoing Example.

11. Perishable material preserved in accordance with the method of any preceding claim.

12. An apparatus suitable for preserving perishable material in accordance with Claim 1, which apparatus comprises a container for accommodating the perishable material, means for introducing a stream of gaseous nitrogen into an upper region of the container, means allowing the escape of the stream of gaseous nitrogen from a lower region of the container, monitoring means for monitoring the humidity of the escaping stream of nitrogen, and means responsive to said monitoring means for controlling the relative humidity of the stream of nitrogen introduced into the container.

13. An apparatus as claimed in Claim 12, substantially as hereinbefore described with reference to, and as illustrated in, Figure 1 of the accompanying drawings.

HASELTINE, LAKE & CO.,
Chartered Patent Agents,
28, Southampton Buildings,
Chancery Lane, London, WC2A 1AT,
—and—
Temple Gate House,
Temple Gate, Bristol BS1 6PT,
9 Park Square, Leeds LS1 2LH.

Printed for Her Majesty's Stationery Office by the Courier Press, Leamington Spa, 1975.
Published by the Patent Office, 25 Southampton Buildings, London, WC2A 1AY, from which copies may be obtained.

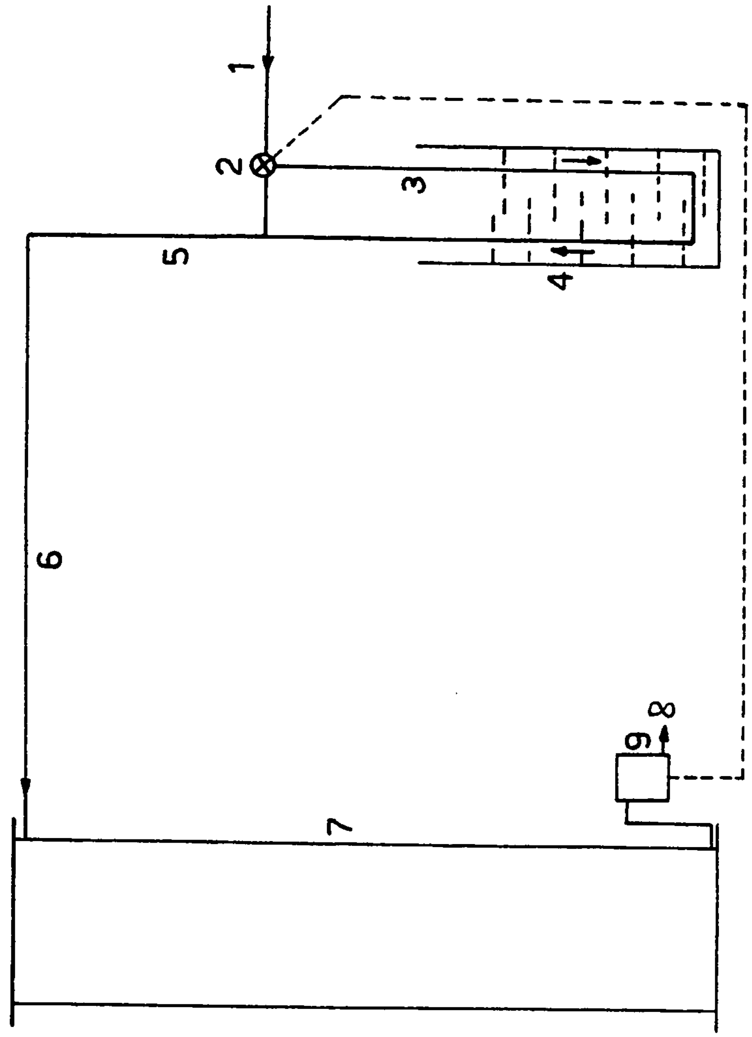


FIG. 1

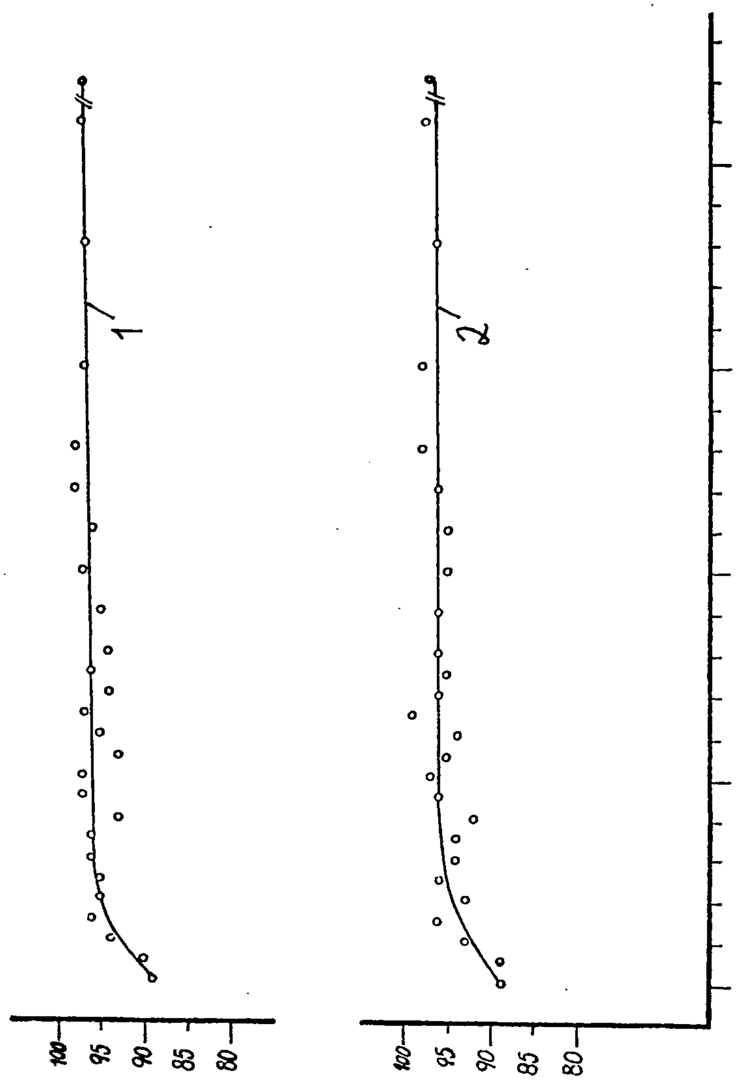


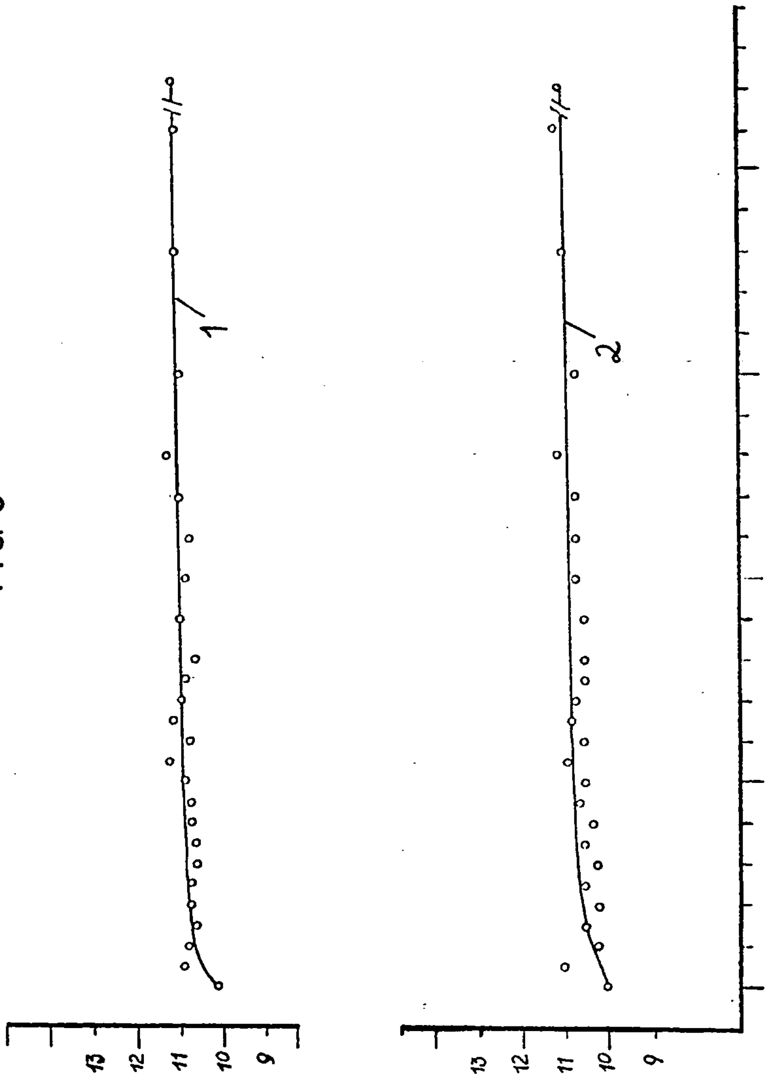
FIG. 2

1379707 COMPLETE SPECIFICATION

4 SHEETS *This drawing is a reproduction of the Original on a reduced scale*

Sheet 3

FIG. 3



1379707 COMPLETE SPECIFICATION

4 SHEETS *This drawing is a reproduction of the Original on a reduced scale*

Sheet 4

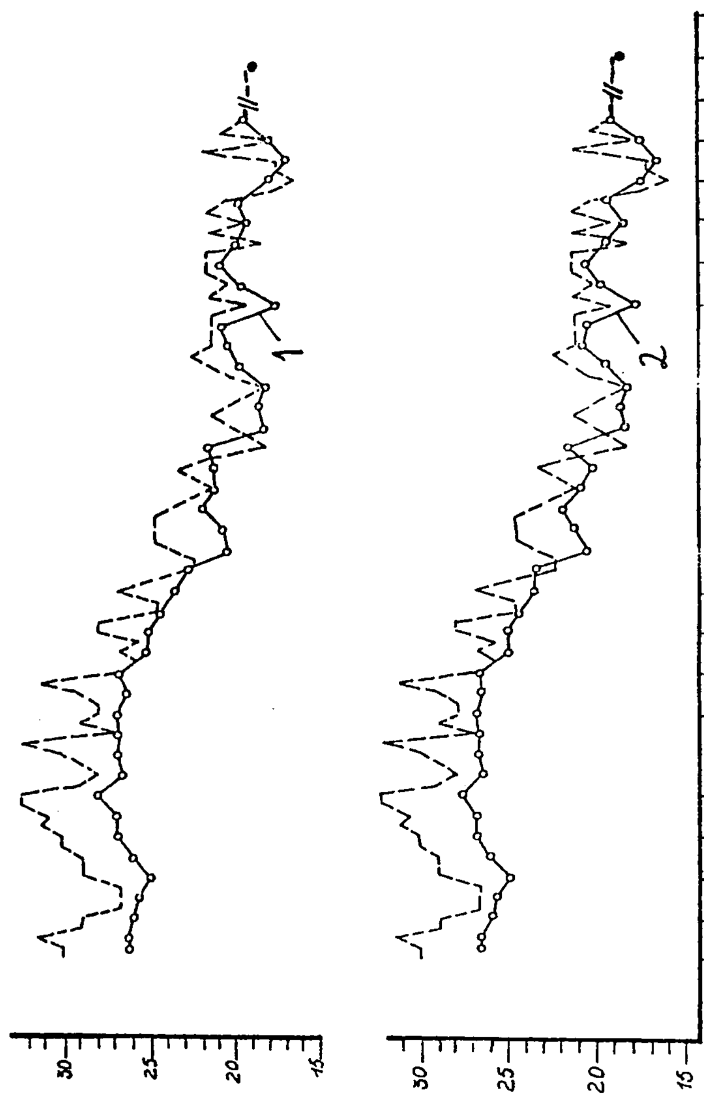


FIG 4